INSTRUCTIONS

FOR THE

MANUFACTURE OF SALTPETRE;

By JOSEPH LeCONTE,

PROFESSOR OF CHEMISTRY AND GEOLOGY IN SOUTH CAROLINA COLLEGE.

PUBLISHED BY AUTHORITY OF THE EXECUTIVE COUNCIL,

UNDER DIRECTION OF

Col. JAMES CHESNUT, Jr.,

CHIEF OF MILITARY DEPARTMENT.

DropBooks

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This pamphlet is issued with the view of supplying information to those who may be inclined to engage in the production of saltpetre.

As the refinement will require a process much more difficult and expensive, the State will undertake that. Private enterprise can thus readily furnish the crude material, which the State will purchase at a fair price, and prepare for all the uses required.

JAMES CHESNUT, JR.

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INSTRUCTIONS.

By the request of the Chief of the Department of the Military, under authority of the Executive Council, I have been induced to publish, for the instruction of planters and manufacturers, a very succinct account of the most approved methods of manufacturing saltpetre. In doing so, I shall aim only at brevity and clearness.

The general conditions necessary to the formation of saltpetre are: 1st, the presence of decaying organic matter, animal or vegetable, especially the former; 2d, an alkaline or earthy base, as potash or lime; 3d, sufficient moisture; 4th, free exposure to the oxygen of the air; and 5th, shelter from sun and rain.

These conditions are often found in nature, as in the soil of *all caves*, but particularly those in limestone countries; and still more frequently under a concurrence of circumstances which, though not strictly natural, is at least accidental, so far as the formation of nitre is concerned, as in cellars, stables, manure-heaps, &c. In crowded cities, with narrow, dirty streets and lanes, the decomposing organic matter with which the soil is impregnated becomes gradually nitrified, oozes through, and dries on the walls and floor of the cellars, as a whitish crust, easily detectible as saltpetre by the taste. The same salt may be found in the soil beneath stables of several years' standing, particularly if lime or ashes have been used to hasten the decomposition of the manure; also in the earth of sheep and cattle pens, if these have remained several years in the same position; also in the soil beneath manure-heaps, particularly if lime or ashes have been added to them, as is common among farmers in making compost. It is very important, then, that the soil of such caves, cellars, stables, pens and manure-heaps, as described above, should be tested for saltpetre. If the salt exists in considerable quantities, it may be detected by the taste; if not, a small quantity of the earth may be leached, and the ley boiled down to dryness, and then tested by the taste. If there be still any doubt, any chemist or educated physician may test it. If the earth contains saltpetre in sufficient quantities, it must be leached, and the salt crystallized, by methods which we have described below.

By these means, if diligently used in all parts of the State, it is hoped that an *immediate* and not inconsiderable amount of saltpetre may be obtained. It is not believed, however, that the supply thus obtained will be sufficient for the exigencies of the war. It is very important, therefore, that steps should be taken to insure a sufficient and *permanent* supply of this invaluable article. This can only be done by means of *nitre-beds*. I proceed, then, to give a very brief account of the method of making these.

NITRE-BEDS.

The most important prerequisite in the construction of nitre-beds in such manner as to yield nitre in the shortest possible time, is a good supply of *thoroughly rotted* manure of the richest kind, in the condition usually called *mould*, or *black earth*. It is believed that in every vicinity a considerable supply of such manure may be found, either ready prepared by nature, or by the farmer and gardener for agricultural and horticultural purposes. To make the *bed*, a floor is prepared of clay, well rammed, so as to be impervious to water. An intimate mixture is then made of rotted manure, old mortar coarsely ground, or wood ashes (leached ashes will do), together with leaves, straw, small twigs, branches, &c. to give porosity to the mass, and a considerable quantity of common earth, if this has not been sufficiently added in the original manure-heap. The mixture is thrown somewhat lightly on the clay floor, so as to form a porous heap four or five feet high, six or seven wide, and fifteen feet long. The whole is then covered by a rough shed to protect from weather, and perhaps protected on the sides in some degree from winds. The heap is watered every week with the richest kinds of liquid manure, such as urine, dung-water, water of privies, cess-pools, drains, &c. The quantity of liquid should be such as to keep the heap always *moist*, but not *wet*. Drains, also, should be so constructed as to conduct any superfluous liquid to a tank, where it is preserved and used in watering the heaps. The materials are turned over to a depth of five or six inches every week, and the whole heap turned over every month. This is not always done, but it hastens very much the process of nitrification. During the last few months of the process, no more urine, nor liquid manure of any kind, must be used, but the heaps must be kept moist by water only. The reason of this is, that undecomposed organic matter interferes with the separation of the nitre from the ley. As the heap *ripens*, the nitre is brought to the surface by evaporation, and appears as a whitish efflorescence, detectible by the taste. When this efflorescence appears, the surface of the heap is removed, to the depth of two or three inches, and put aside under shelter, and kept moist with water. The nitre contained is thus considerably increased. When the whitish crust again appears, it is again removed until a quantity sufficient for leaching is obtained. The small mound which is thus left is usually used as the nucleus of a new heap. By this method it is believed that an abundant supply of nitrified earth, in a condition fit for leaching, may be obtained by autumn or early winter.

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I have spoken thus far of the method of preparing a single heap, or nitre-bed, such as any farmer or gardener may prepare with little trouble. But where saltpetre is manufactured on a large scale, as in the *saltpetre plantations*, many such beds are made and symmetrically arranged, so as to economize space; all under the same roof, with regularly arranged drains, all leading to a large cistern. In such plantations everything may be carried on with more economy, and with correspondingly increased profits.

PREPARATION OF MOULD.

I have supposed that there is already a considerable supply of rotted manure, prepared for other purposes, in a condition fitted for making nitre-beds; but after the

present year this precarious supply must not be relied on. Systematic preparation of mould or black earth must be undertaken. The process of preparation is so precisely similar to that of compost manure that little need be said, the chief difference being the greater richness in nitrogenous matter in the case of compost intended for nitre-beds. First prepare a floor of well-rammed clay; on this place a layer of common soil, mixed with broken old mortar or ashes, six or eight inches thick; then a layer of vegetable matter -- straw, leaves, rank weeds, &c. then a layer of animal matter, dung, flesh, skin, scrapings of drains, sinks, &c. then another layer of mixed earth and mortar or ashes, and so on until a heap six feet high is made. Brush and sticks are often introduced, also, to increase the porosity of the mass. The whole is protected from the weather, and watered every week or two with urine or dung-water, until the organic matter is entirely decomposed into a black mass. This will take place in about a year, or perhaps less, in our climate. The whole is thoroughly mixed, and is then fit for making nitre-beds, as already explained.

Thus it is hoped that the preparation of saltpetre may be set on foot at once in three different stages of advance, viz.: by the collection of already nitrified earth; by the making of nitre-beds from already formed black earth; and by the preparation of black earth. By leaching, the first would yield immediate results, the second in six or eight months, and the last in about eighteen months or two years.

The method I have given above is that of the French. Other methods are precisely the same in principle, and differ only slightly in some of the details.

PRUSSIAN METHOD.

Five parts of black earth and one of spent ashes or broken mortar are mixed with barley straw, to make the mass porous. The mixture is then made into heaps six feet high and fifteen feet long with one side perpendicular (and hence called walls), and the opposite side sloping regularly by a series of terraces or steps. Straight sticks are generally introduced, and withdrawn when the mass is sufficiently firm. By this means air and water are introduced into the interior of the mass. The heap is lightly thatched with straw, to protect from sun and rain. The whole is frequently watered with urine and dung-water. The perpendicular side being turned in the direction of the prevailing winds, the evaporation is most rapid on that side. The liquid with which the heap is watered is drawn by capillarity and evaporation to this side, carrying the nitre with it, and the latter effloresces there as a whitish crust. The perpendicular wall is shaved off two or three inches deep as often as the whitish incrustation appears, and the material thus removed is kept for leaching. The leached earth, mixed with a little fresh mould, is thrown back on the sloping side of the heap, and distributed so as to retain the original form of the heap. Thus the heaps slowly change their position, but retain their forms. This method yields results in about a year-- probably in our climate in eight months.

SWEDISH METHOD.

Every Swede pays a portion of his tax in nitre. This salt is therefore prepared by almost every one on a small scale. The Swedish method does not differ in any essential respect from those I have already described. First a clay floor; upon this is placed a mixture of earth, mould, spent ashes, animal and vegetable refuse of all kinds. Small twig branches, straw and leaves are added, to make the mass porous; a light covering, to protect from weather, frequent watering with urine or dung-water, and turning over every week or two. The process is precisely the same as the French, except that the process of *preparation* and *nitrification* are not separated. I only mention it to show that nitre may be made by every one on a small scale. By this method the beds are ripe in two years--perhaps in less time in this country.

SWISS METHOD.

The method practiced by the small farmers in Switzerland is very simple, requires little or no care, and is admirably adapted to the hilly portions of our State.

A stable with a board floor is built on the slope of a hill (a northern slope is best), with one end resting on the ground, while the other is elevated, several feet, thus allowing the air to circulate freely below. Beneath the stable a pit, two or three feet deep, and conforming to the slope of the hill, is dug and filled with porous sand, mixed with ashes or old mortar. The urine of the animals is absorbed by the porous sand, becomes nitrified, and is fit for leaching in about two years. The exhausted earth is returned to the pit, to undergo the same process again. This leached earth induces nitrification much more rapidly than fresh earth; so that after the first crop the earth may be leached regularly every year. A moderate-sized stable yields with every leaching about one thousand pounds of saltpetre.

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LEACHING.

When the process of nitrification is complete, the earth of the heaps must be leached. Manufacturers are accustomed to judge roughly of the amount of nitre in any earth by the taste. A more accurate method is by leaching a small quantity of the earth, and boiling to dryness, and weighing the salt. There is much diversity of opinion as to the per centage of nitre necessary to render its extraction profitable. The best writers on this subject vary in their estimates from fifteen pounds to sixty pounds of salt per cubic yard of nitrified earth. The high price of nitre with us at present would make a smaller per centage profitable. This point, however, will soon be determined by the enterprising manufacturer.

In the process of *leaching*, in order to save fuel, we must strive to get as strong a solution as possible, and at the same time to extract all or nearly all the nitre. These two objects can only be attained by repeated leachings of the same earth, the ley thus obtained being used on fresh earth until the strength of the ley is sufficient. A quantity of nitrified

earth is thrown into a vat, or ash-tub, or barrel, or hogshead with an aperture below, closely stopped and covered lightly with straw. Water is added, about half as much in volume as the earth. After stirring, this is allowed to remain twelve hours. Upon opening the bung, about half the water runs through containing, of course, one-half the nitre. Pure water, in quantity half as much as first used, is again poured on, and after a few moments run through. This will contain one-half the remaining nitre, and therefore one-fourth of the original quantity. Thus the leys of successive leachings become weaker and weaker, until, after the sixth leaching, the earth is considered as sufficiently exhausted. The exhausted earth is thrown back on the nitre-beds, or else mixed with black earth to form new beds. The leys thus obtained are used upon fresh earth until the solution is of sufficient density to bear an egg. It then contains about a pound of salt to a gallon of liquid.

CONVERSION.

The ley thus obtained contains, besides nitrate of potash (nitre), also nitrate of lime and magnesia, and chlorides of sodium and potassium. The object of the next process is to convert all other nitrates into nitrate of potash. This is done by adding wood ashes. The potash of the ashes takes all the nitric acid of the other nitrates forming the nitrate of potash (nitre), and the lime and magnesia are precipitated as an insoluble sediment. Sometimes the ashes is mixed with the nitrified earth and leached together, sometimes the saltpetre ley is filtered through wood ashes, sometimes the ley of ashes is added to the saltpetre ley. In either case the result is precisely the same.

CRYSTALLIZATION.

The ley thus converted is then poured off from the precipitate, into copper or iron boilers. It still contains common salt (chloride of sodium) in considerable, and some other impurities in smaller, quantities. It is a peculiarity of nitre, that it is much *more* soluble than common salt in boiling water, but much less soluble in cold water. As the boiling proceeds, therefore, and the solution becomes more concentrated, the common salt is, most of it, precipitated in small crystals, as a sandy sediment, and may be raked out. Much organic matter rises as scum, and must also be removed. When the concentration has reached almost the point of saturation, the boiler must be allowed to cool. This is known by letting fall a drop of the boiling liquid upon a cold metallic surface; if it quickly crystallizes, it is time to stop the boiling. It is now poured into large receivers and left to cool. As the ley cools, nearly the whole of the nitre separates in the form of crystals, which sink to the bottom. These are then removed, drained by throwing them in baskets, and dried by gentle beat. The mother-liquor is either thrown back into the boilers, or else used in watering the heaps. The product thus obtained is the *crude saltpetre* of commerce. It still contains fifteen to twenty-five per cent. of impurities, principally common salt (chloride of sodium), chloride of potassium and organic matter. In this impure form it is usually brought to market.

There is still another process, viz: that of *refining*, by which the whole of the impurities is removed. This is seldom done by the manufacturer, but by a separate class, called the refiners.

REFINING.

One hundred gallons of water is poured into a boiler, and crude saltpetre added from time to time, while the liquid is heating, until four thousand pounds are introduced. This will make a saturated solution of nitre. The scum brought up by toiling must be removed, and the undissolved common salt scraped out. About sixty gallons cold water is now added gradually, so as not to cool the liquid too suddenly. From one to one and a-half pounds of glue, dissolved in hot water, is added, with stirring. Blood is sometimes used instead of glue. The glue seizes upon the organic matter, and they rise together as scum, which is removed. Continue the boiling until the liquid is clear. The liquid is then suffered to cool to one hundred and ninety-four degrees, and then carefully ladled out into the crystallizers. These are large shallow vats, with the bottom sloping gently to the middle. In these the cooling is completed, with constant stirring. In the process of cooling nearly the whole of the nitre is deposited in very fine, needle-like crystals, which, as they deposit, are removed and drained. In this condition it is called *saltpetre flour*. The object of the constant stirring is to prevent the aggregation of the crystals into masses, from which it is difficult to remove the adhering mother-liquor. The saltpetre flour is then washed of all adhering mother-liquor. For this purpose it is thrown into a box with a double bottom; the lower bottom with an aperture closely plugged, and the false bottom finely perforated. By means of a watering pot a *saturated solution of pure nitre* is added, in quantity sufficient to moisten thoroughly the whole mass. After remaining two or three hours to drain, the plug is removed and the solution run out. This is sometimes repeated several times. The saturated solution of nitre cannot, of course, dissolve any more nitre, but dissolves freely the impurities present in the adhering mother-liquor. Last of all, a *small quantity of pure water*-- only about one pound to fifty-three pounds of the nitre to be washed-- is added in the same manner, and run off at the end of two hours. The nitre is now dried by gentle heat and constant stirring, and may be considered quite pure, and fit for the manufacture of gunpowder.

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ANALYSIS.

As the value of crude saltpetre depends upon the quantity of pure nitre which it contains, it is important to give some simple methods of estimating its purity:

1. The first method is founded upon the fact, already alluded to, that a saturated solution of any salt will not dissolve any more of that salt, but will freely dissolve other salts. Twelve ounces of crude saltpetre is well ground, and twelve ounces of a saturated solution of pure nitre added. The mixture is stirred fifteen minutes, allowed to settle, and the *liquid* carefully poured off. Six to nine ounces more of the saturated solution of nitre

is again poured on, the mixture stirred ten minutes, and the *whole* thrown on a filter, and allowed to remain until thoroughly drained. The filter, with its contents, is then pressed upon blotting paper, or slab of plaster, or other absorbent substance-- the nitre carefully removed and dried, and carefully weighed. The loss of weight indicates the impurity originally present in the crude saltpetre. About two per cent. should be deducted from the estimate of impurity, or added to the estimate of pure nitre; since, although a saturated solution of nitre will not dissolve any more *pure* nitre, still, if any common salt be present, a small additional quantity of nitre is taken up.

2. Another method of estimating saltpetre is founded upon the fact that nitre mixed with charcoal and heated is entirely converted into carbonate of potash, while common salt is not affected. If the saltpetre be mixed with charcoal alone, the reaction is apt to be violent and explosive. To moderate the violence of the action, the saltpetre must be largely mixed with common salt, which does not interfere with the reaction. One part crude saltpetre, four parts common salt, and one-half part charcoal, are mixed and thrown gradually in a red-hot crucible, or else heated in an iron spoon, until reaction ceases. The whole of the nitre is now changed into carbonate of potash, which may be dissolved in water and filtered. The solution thus obtained, being alkaline may be estimated by the quantity of sulphuric or other acid of known strength necessary to completely neutralize it. This is done by means of the instrument called the alkalimetre. One part of pure potassa corresponds to 2.14 parts of nitre; or one part carbonate potassa corresponds to 1.46 parts nitre. The objection to this method is, that it requires the use of the alkalimetre; and, therefore, a degree of care and an amount of accuracy which can hardly be expected in practical men.

3. The third method of estimation depends upon the fact that a strong hot solution of nitre crystallizes on cooling, and that the temperature at which crystals *begin* to deposit (or point of saturation) depends upon the amount of nitre present in the solution, irrespective of the presence of impurities. In one hundred parts of hot water is dissolved forty parts of crude saltpetre. A very delicate thermometer is introduced, the liquid allowed to cool slowly, and the temperature at which crystals *begin* to deposit is accurately observed. The higher the temperature, the larger the quantity of nitre present in the solution, and, therefore, the purer the saltpetre. Tables have been constructed giving the saturating point for solutions containing different quantities of nitre.

I have constructed, from materials derived from the best French authorities, a table which is sufficiently complete and accurate for all practical purposes.

In a saturated solution of nitre, one hundred parts by weight of water at;

- 32° contains 13.32 parts of nitre.

- 33° contains 13.64 parts of nitre.
- 34° contains 13.97 parts of nitre.
- 35° contains 14.31 parts of nitre.
- 36° contains 14.66 parts of nitre.
- 37° contains 15.02 parts of nitre.
- 38° contains 15.40 parts of nitre.
- 39° contains 15.79 parts of nitre.
- 40° contains 16.19 parts of nitre.
- 41° contains 16.50 parts of nitre.
- 42° contains 16.91 parts of nitre.
- 43° contains 17.33 parts of nitre.
- 44° contains 17.76 parts of nitre.
- 45° contains 18.20 parts of nitre.
- 46° contains 18.66 parts of nitre.
- 47° contains 19.13 parts of nitre.
- 48° contains 19.61 parts of nitre.
- 49° contains 20.10 parts of nitre.
- 50° contains 20.60 parts of nitre.
- 51° contains 21.12 parts of nitre.
- 52° contains 21.65 parts of nitre.
- 53° contains 22.20 parts of nitre.
- 54° contains 22.76 parts of nitre.
- 55° contains 23.23 parts of nitre.
- 56° contains 23.81 parts of nitre.
- 57° contains 24.40 parts of nitre.
- 58° contains 25.00 parts of nitre.
- 59° contains 25.60 parts of nitre.
- 60° contains 26.21 parts of nitre.
- 61° contains 26.82 parts of nitre.
- 62° contains 27.44 parts of nitre.
- 63° contains 28.07 parts of nitre.
- 64° contains 28.70 parts of nitre.
- 65° contains 29.34 parts of nitre.
- 66° contains 30.09 parts of nitre.
- 67° contains 30.74 parts of nitre.
- 68° contains 31.40 parts of nitre.
- 69° contains 32.08 parts of nitre.
- 70° contains 32.77 parts of nitre.
- 71° contains 33.48 parts of nitre.
- 72° contains 34.20 parts of nitre.
- 73° contains 34.94 parts of nitre.
- 74° contains 35.69 parts of nitre.
- 75° contains 36.46 parts of nitre.

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- 76° contains 37.25 parts of nitre.
- 77° contains 38.05 parts of nitre.
- 78° contains 38.85 parts of nitre.
- 79° contains 39.65 parts of nitre.
- 80° contains 40.46 parts of nitre.
- 81° contains 41.27 parts of nitre.
- 82° contains 42.09 parts of nitre.
- 83° contains 42.92 parts of nitre.
- 84° contains 43.76 parts of nitre.
- 85° contains 44.62 parts of nitre.
- 86° contains 45.50 parts of nitre.
- 87° contains 46.42 parts of nitre.
- 88° contains 47.33 parts of nitre.
- 89° contains 48.26 parts of nitre.
- 90° contains 49.20 parts of nitre.
- 91° contains 50.16 parts of nitre.
- 92° contains 51.13 parts of nitre.
- 93° contains 52.11 parts of nitre.
- 94° contains 53.10 parts of nitre.
- 95° contains 54.10 parts of nitre.

By comparing the quantity of *pure nitre*, as determined by inspection of the table, with the quantity of *crude saltpetre* dissolved, the per centage of pure nitre may be easily calculated. Thus, if crystals begin to deposit at 68°, the quantity of nitre contained in a hundred parts of water is 31.40 parts; dividing this by 40 parts crude nitre, originally dissolved, gives 76 per cent. of pure nitre in the sample examined. In the foregoing example I have used 40 parts crude saltpetre; but we are by no means limited to this number. On the contrary, in our climate a larger quantity, as 50, or even 60, parts is preferable. For it will be observed that at 80° more than 40 parts of nitre are soluble in 100 parts of water, and that, therefore, in our summer weather, if only 40 parts of crude saltpetre are used in the experiment, artificial cold will be necessary to produce crystallization. To avoid this inconvenience, it is only necessary to use a larger proportion of crude saltpetre in the experiment. Thus, if 50 parts are used, and crystallization commences at 80°, the quantity of pure nitre, by the table, being 40.46, the per centage is 40.46 / 50 = 80.9. For higher summer temperature, it will be, of course, necessary to use a still larger quantity of crude saltpetre in the experiment. This method has the advantage of great ease and rapidity of execution.

In conclusion, a word by way of encouragement to manufacturers in undertaking this work.

It will be seen that under the most favorable circumstances saltpetre cannot be made in any considerable quantity in less than six or eight months, and that if we commence now the preliminary process of *preparing black earth*, so as to insure a sufficient and permanent supply, results cannot be expected under eighteen months or two years. Let no one be discouraged by this fact, under the idea that the war *may* not last so long, and all their work *may* be thrown away. There is every prospect now of the war continuing at least several years, and of our being thrown entirely on our own resources for war materials. Besides, even if the war should be discontinued, the work is by no means lost. The method of preparing and making saltpetre-beds is precisely the most approved method of making the best manure, and all the labor and pains necessary for the *preparation of black earth*, and the construction of saltpetre-beds, and which I hope to induce my fellow-countrymen to undertake under the noble impulse of patriotism, ought to be annually undertaken by every planter, under the lower impulse of a wise self-interest, and would be amply rewarded in the increased production of field crops.

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